U. S. NAVAL ORDNANCE TEST STATION
CHINA LAKE, CALIFORNIA

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From: Commander, U. S. Naval Ordnance Test Station
To: Initial Distribution List of NAVWEPS REPORT 7764, Part 1

Subj: NAVWEPS REPORT 7764, Part 1, "Standard Laboratory Procedures Part 1. Stability, Sensitivity, Physical Properties, and Analyses for High Explosives" dated August 1961; transmittal of errata sheet for

Encl: (1) Errata sheet dated 11 January 1962

1. It is requested that the corrections noted on the enclosed errata sheet be incorporated in subject report.

C. E. VAN HAGAN
By direction

ERRATA

Page 4, line 7; ρ liquid = $2.523 \frac{Wax}{Wax - Wlx}$ should be

$$\rho \text{ liquid} = 2.523 \div \frac{Wax}{Wax - Wlx}$$

Page 12, paragraph under "Addendum" should be changed to read:

line 3: ". . .and a mean established. When. . ."
line 6: ". . .and/or corrected. At least. . ."

Page 13, the fifth line column 5; 1.7^- should be 1.7^+
the fifth line column 6; 1.6 should be 1.6^-.

Page 13, under paragraph which starts "In formula (1) below, line 4

(1) $m = c + d \frac{A}{N} \pm 1/2$ should be (1) $M = c + d \left(\frac{A}{N} \pm 1/2\right)$

line 6 (2) s.d. = $1.62\, d \frac{NB - A^2}{N^2} + 0.029$ should be

$$\text{s.d.} = 1.62\, d \left(\frac{NB - A^2}{N^2}\right) + 0.029$$

Page 15, line 15; $V_\mu = \frac{273.2\,(P_a + d - k)}{76\,w} \frac{V_t - V_s}{t_o} + \frac{V_c + d\,L}{t_r}$ should be

$$V_\mu = \frac{273.2\,(P_a + d - K)}{76\,w} \left[\frac{V_t - V_s}{t_o} + \frac{V_c + d\,L}{t_r}\right]$$

Page 16, Fig. 3; On the left hand side of the page the V_c with the arrow leading in should be V_t.

Page 17, paragraph 2; "Sample Specifications", line 5; (70 to 80°C) should be (70 to 80°F).

11 January 1962
Enclosure 1

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Part 1
NOTS TP-2742
COPY 68

STANDARD LABORATORY PROCEDURES

Part 1. Stability, Sensitivity, Physical Properties, and Analyses for High Explosives

by

O. K. Pennington
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ABSTRACT. The Standard Laboratory Procedures for explosives herein reported are those developed for use by the Explosives and Pyrotechnics Division of the Propulsion Development Department. This part contains a portion of the procedures used at the Naval Ordnance Test Station and will be followed by Part 2, Determination of Explosives Properties And Parameters Relating Especially to Plastic-Bonded Explosives, at a future date.

U.S. NAVAL ORDNANCE TEST STATION

China Lake, California

August 1961

U. S. NAVAL ORDNANCE TEST STATION

AN ACTIVITY OF THE BUREAU OF NAVAL WEAPONS

C. BLENMAN, JR., CAPT., USN
Commander

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Technical Director

FOREWORD

This compilation consists of a series of procedures as used by the Naval Ordnance Test Station in characterizing explosives and ingredients of explosives. It is Part 1 of a series of standard procedures to be issued for propellants and explosives and is issued as a NAVWEPS publication because of requests for copies of the first manuscript issued informally January 1958. Most of the procedures have been modified and rewritten since that time and the revised procedures are given here.

Much of this work was sponsored by RUUO under Task Assignment RUUO-3E-015/216-1/F008-10-004 and Local Project 992.

These procedures have been reviewed by K. S. Skaar and H. J. Gryting.

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Released under
authority of:

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INTRODUCTION

Explosives programs at the Naval Ordnance Test Station have been conducted since 1945. Early work was done under sponsorship of the Atomic Energy Commission. Since 1954 work has been sponsored by the Bureau of Naval Weapons. A series of procedures came into use for characterizing explosives and ingredients for them. These are tabulated here for use by personnel who may prepare or use explosives compositions.

DENSITY DETERMINATION BY MEASURING METHOD

THEORY

A measured or calculated density is determined on regular specimens by measuring their dimensions and dividing their volume into their weight. The method is faster and easier than the immersion method for regular samples and although there is some error in measurement, the results are comparable to those obtained by the immersion method.

SAMPLE SPECIFICATIONS

1. The sample shall be free from cracks and chips, and shall have parallel ends and straight sides as determined by measurements.

2. The sample container and samples within shall be clearly marked with proper identification.

EQUIPMENT AND SUPPLIES NEEDED

1. Micrometer which is read to 0.001 inch and has a ratched adjustment.

2. Analytical balance which is capable of being read to at least 0.0002 gram.

PROCEDURE

1. At least three height measurements are made using a micrometer and the average recorded. Measure center and edges in two diametrically opposed positions.

2. Four diameter measurements are made using a micrometer and the average recorded. Take measurements 90° apart, 1/4 distance from each end.

3. Weigh the sample to the nearest 0.0001 or 0.0002 grams.

CALCULATIONS

1. The measured density is calculated by

$$\rho_m = \frac{g}{\text{vol.}} = \frac{g}{r^2h \text{ (in cm)}} = \frac{(0.07770)\text{grams}}{d^2h \text{ (in inches)}}$$

where ρ_m = density
g = weight in grams
r = radius of pellet
h = height of pellet
d = diameter of pellet

2. Factor tables and nomographs are available for standard size samples of standard composition batches. These tables cover sample sizes of diameters 0.497 - 0.502-inch and height combinations ranging from 0.670 - 0.885-inch.

3. Percentage maximum theoretical density is calculated by assuming complete additivity by volume of components and dividing the measured density by the maximum theoretical density and multiplying by 100.

DENSITY DETERMINATION BY IMMERSION METHOD

THEORY

The immersion density method, based on Archimedes' principle, or the loss-in-weight-in-liquid method is used on irregularly shaped samples. The results are comparable to those from the measured density method when porosity is not a problem.

SAMPLE SPECIFICATIONS

1. The samples for immersion density shall be of a size and weight which can be easily handled using the 200 gram or 2,000 gram balances. Larger scale samples are not run by this laboratory.

EQUIPMENT AND SUPPLIES NEEDED

1. Analytical balance which can be read to at least 0.0002 gram. (Use a projection beam type if possible with 100 mg on its scale.)

2. Beryllium file used to remove water-leached surfaces if necessary.

3. 250 ml beaker or other suitable size filled with 0.1% water solution of aerosol or other suitable solution.

4. Brass bridge to support the beaker over the balance pan.

5. Copper wire basket and tare counterweight.

PROCEDURE

1. Place the filled beaker on the balance floor to allow solution to reach room temperature.

2. If necessary, file (behind an adequate shield) any water-leached surfaces and any rough spots with a beryllium file catching powder filings in a porcelain pan. These filings are then transferred to the scrap container. Immediately replace number on sample if number was removed. Do not file new materials until permission has been received.

3. Wipe all samples with a cloth and check all sample numbers.

4. Weigh the samples in air to 0.0002 grams.

5. Weigh a piece of glass whose density has been standardized against calcite crystals. The standard glass pieces have a density of 2.523 g/cc.

6. Place the brass pan over the left balance pan and place the filled beaker on it. Suspend the wire basket from the lower balance hook so that it is not touching the beaker at any point. Place the tare counterweight on the right balance pan.

7. Re-zero the balance.

8. Immerse samples individually in the liquid and determine the wet weight of each as quickly as possible. Make sure that no bubbles adhere to the sample or basket.

9. Immerse the glass and determine its wet weight. There should be a glass wet-weight taken every 10-12 samples and taken more often if a rise in temperature is suspected. The glass wet-weight should be taken in the middle of a series of weights so as to get the average density of the liquid over the series.

10. If some of the samples appear very porous the earliest possible wet weight is advisable.

CALCULATIONS

1. The calculations for immersion density is as follows:

$$\rho_I = \frac{W_a}{W_a - W_l} \times \rho \text{ liquid}$$

where ρ_I = immersion density
W_a = weight in air of material whose density is determined
W_l = weight in the liquid
ρ liquid = density of the liquid

2. The density of the liquid is calculated as follows:

$$\rho \text{ liquid} = 2.523 \frac{Wax}{Wax - Wlx}$$

where ρ liquid = density of the liquid
2.523 = density of the glass
Wax = weight of the glass
Wlx = weight of the glass when immersed in liquid.

3. Checks are made on "out of line" results and the maximum and minimum in a series of about 12 samples. The checks should agree with ± 1 in the last calculated decimal place.

Standard values for theoretical maximum densities to be used in the preceding procedures are:

Materials	Density, g/cm^3	Remarks
RDX	1.816	Normal Holston RDX containing some HMX
HMX	1.90	Normal Holston 98% HMX[1]
Nylon	1.13	Zytel 61 or 63
Aluminum	2.70	Considers impurities including some Al_2O_3
TNT	1.654	

PERCENTAGE GREASE IN ALUMINUM POWDERS

THEORY

The percentage grease is determined by dissolving the metal and collecting the remaining grease in a tared beaker.

SAMPLE SPECIFICATIONS

No specific requirements.

[1] Solubility Properties of RDX and HMX, by Herring and Beard. Holston Defense Corporation. 3 Oct 1952. Control #20T-3-Series A.

EQUIPMENT AND SUPPLIES NEEDED

1. Analytical balance accurate to 0.0002 grams
2. 400 ml beakers and watch glasses to fit
3. 100 ml beakers
4. Small funnels
5. Filter rack or stand
6. 100 ml graduate
7. Distilled water
8. 6 N HCL
9. 1 N HCL
10. Glass stirring rods
11. Hot plate

PROCEDURE

1. Weigh 2 ± 0.0002 grams of metal powder into a 400 ml beaker.

2. Add 10 ml hot distilled water and stir with a glass stirring rod until wet. (If difficulty is encountered, add 1-5 ml of 1 N HCL and stir.)

3. Add 90 to 100 ml boiling distilled water and cover with a watch glass.

4. Add, drop by drop at first while stirring, 6 N HCL. Keep the reaction at a slow rate. Excess heat causes the hydroxide to be formed, thus ruining the test.

5. When the reaction is complete, as seen by absence of metal, allow the beaker to cool.

6. Pour the solution through a Whatman 50 or 52 filter paper; discard the filtrate.

7. Wash residual materials from the 400 ml beaker, stirring rod, and watch glasses onto the filter paper using distilled water.

8. Dry the filter paper in air.

9. Wash the filter paper, original beaker, stirring rod, and watch glasses with acetone collecting the filtrate in a tared 100 ml beaker.

10. Evaporate the washing to dryness on a steam bath and dry the beaker in an oven at 100°C for one hours.

11. Weigh back the beaker with its grease inner coating.

CALCULATIONS

$$\text{Percent grease} = \frac{(\text{Wt beaker and grease}) - \text{Wt beaker}}{\text{Wt sample}} \times 100$$

DETERMINATION OF COMPOSITION OF COMPOSITION B AND CYCLOTOL BY USE OF SELECTIVE SOLVENTS

THEORY

The method given here is a general one which can be adapted to most formulations of inert or explosive samples. By this method the components are successively extracted out using selective solvents. The specific method for Composition B and Cyclotol 75/25 are given. Reference is made to a method for HEX-1.

SAMPLE REQUIREMENTS

1. Five gram samples of powder or raw material are used.

2. A cast or pressed sample cannot exceed 1 inch in diameter and should be no longer than 1 inch in length.

EQUIPMENT AND SUPPLIES NEEDED

Because this is a general method no list is given. Each specific method would have a specialized list of equipment and supplies.

BRIEF GENERAL EXTRACTION PROCEDURE

1. The sample is weighed into a wide mouth Erlenmeyer flask covered with a specified amount of the first selective solvent.

2. When the sample is broken down and the first ingredient dissolved the remaining sample is washed into a tared crucible.

3. The crucible and residue is dried and weighed and the percent residue calculated.

4. This procedure is repeated working with the residue from the first extraction.

PROCEDURE FOR COMPOSITION B AND CYCLOTOL

1. Weigh either 5 grams of raw material or a cast sample into a 125 ml wide mouthed Erlenmeyer flask.

2. Prepare RDX saturated toluene by stirring 100 grams of RDX in 5 gallons of toluene for 4 to 8 hours at room temperature. Add 25 ml of RDX saturated toluene for a 3 to 5 gram sample, 30 ml. of saturated toluene for a 5 to 8 gram sample and 35 ml of saturated toluene for an 8 to 10 gram sample.

3. Cover the beaker with a Sanitab cap and place on the steam bath stirring occasionally until the sample is completely broken down (about 1/2 hour).

4. Place the beakers in a special tray on an air driven shaker which simulates the swirling motion of the hand for one hour.

5. Transfer the sample to a tared 30 ml capacity medium porosity fritted glass crucible and wash with about 100 ml of RDX saturated benzene (prepared the same way as the RDX saturated toluene) in four or five portions.

6. Dry the crucible at 80°C for 20-30 minutes, cool, weigh the crucible plus residue.

CALCULATIONS

$$\% \text{ RDX} = \frac{(\text{wt crucible + residue}) - \text{wt crucible}}{\text{wt sample}} \times 100$$

% TNT + wax is determined by difference.

HBX-1

HBX-1 can be analyzed by using the procedure listed in NAVORD Report 5014 (NOTS 1350), "An Experimental Analysis for HBX-1," by K. B. Clifton.

SEPARATION AND DETERMINATION OF WAX IN COMPOSITION B

This method determines all the desensitizer in the Composition B. No correction factor is necessary. Like the other analytical procedures in use, the method and equipment is chosen to promote efficiency in handling a group of samples.

This method is based on selective solvents which first extract the TNT and desensitizer, and then remove the TNT. Results are readily reproducible.

1. Equipment

Beakers - 100 ml
Beaker tray
Funnel, fritted glass, medium, 150 ml capacity
Acetic acid - 70%
Carbon tetrachloride, chloroform mixture 60-40% by volume.

2. Procedure

Into a tared beaker weigh exactly 5 grams of the crushed material. Filter through the fritted glass funnel and wash with three ml portions of the solvent mixture. Discard the residue (Caution: Explosive), and evaporate the combined filtrates until the TNT begins to crystallize. The volume will be about 10 ml at this point.

Add 50 ml of 70% acetic acid, warm on the steam bath until all TNT is dissolved. Cool in cold water, then in ice water. The wax will separate and largely adhere to the walls of the beaker.

It is necessary to watch carefully that the material is not cooled fast enough to cause TNT separation, as this will make an extra extraction necessary.

Filter through a clean fritted glass funnel retaining as much solid material as possible in the beaker. Save the funnel as is.

Repeat the extraction of the material in the beaker using 50 ml of 70% acetic acid, heating to dissolve or melt all material, and cooling as above.

Filter through the same funnel as before washing beaker and funnel with 30 ml of distilled water. Rinse funnel with 5 ml of cold acetone.

Wash all wax on the funnel into the original tared beaker using a small quantity of the carbon tet-chloroform mixture.

Evaporate to dryness on the steam bath and dry in oven to constant weight.

Caution: Carbon tet-chloroform vapors are dangerous to health. Do all evaporating under the hood.

Calculate:

$$\% \text{ wax} = \frac{\text{wt of wax in beaker} - \text{wt of beaker}}{\text{Weight of sample}} \times 100$$

IMPACT SENSITIVITY TEST WITH THE 2-1/2 KG HAMMER AND TYPE 12 TOOLS USING PELLETIZED SAMPLES

DESCRIPTION OF THE IMPACT MACHINE

The instrument is a drop-test machine which consists of a mechanism for dropping a weight through a chosen distance upon appropriate tools. It allows a free fall of the weight by means of a electromagnetic weight release; it has three rail guides to control the motion of the weight. The firing tools are an anvil which is held rigidly on the bed plate by an anvil holder and a steel striker moving in an appropriate plunger guide. Both are made of hardened steel. The NOTS machine of the ERL, Bruceton type, has a 337 cm maximum drop height and is equipped with "type 12" tools. The anvil is 1-1/4-inch in diameter and 1-1/4-inch high, the striker is 1-1/4-inch in diameter and 6-1/4-inch long. The top of the striker is slightly rounded with a radius of curvature of 2-1/2-in. The tools are made from Ketos oil hardened tool steel with a hardness between 55-60 c Rockwell. The weight used is 2-1/2 Kg. (Reference 1.)

To tell whether or not an explosion occurs, a microphone (Dynamic, Model 999 made by Turner Co., Cedar Rapids, Iowa) is connected to a peak reading amplifier or noisemeter. By zeroing out room noise and setting the hammer a predetermined height to fall upon an inert sample, a reading is taken upon the dial. In all tests, a reading of at least one or more divisions over the predetermined height is classed as an explosion.

TURNING ON THE MACHINE AND SETTING THE LISTENING DEVICE PRIOR TO TESTING

1. Turn on the switch located at the right of the machine to activate the electromagnet that holds the hammer in position. (See Fig. 1.)

2. Turn on switch No. 1 located on the electronic control box.

3. Push toggle switch No. 2 down and release. The hand on the dial will return to near zero.

4. Turn switch No. 3 clockwise as far as it will go and leave in this position.

5. To zero out room noise adjust the needle of the dial to zero by turning switch No. 4 to the right or left and frequently pushing down toggle switch No. 2 to eliminate the accumulated noise reading.

6. Clean striker and base with acetone and wipe dry with cloth wrapped on spatula at least once every five hammer drops. Clean it so no explosive is retained after every drop using a spatula.

7. Prepare several 35 mg pellets of inert powder. (See Preparation of Sample.)

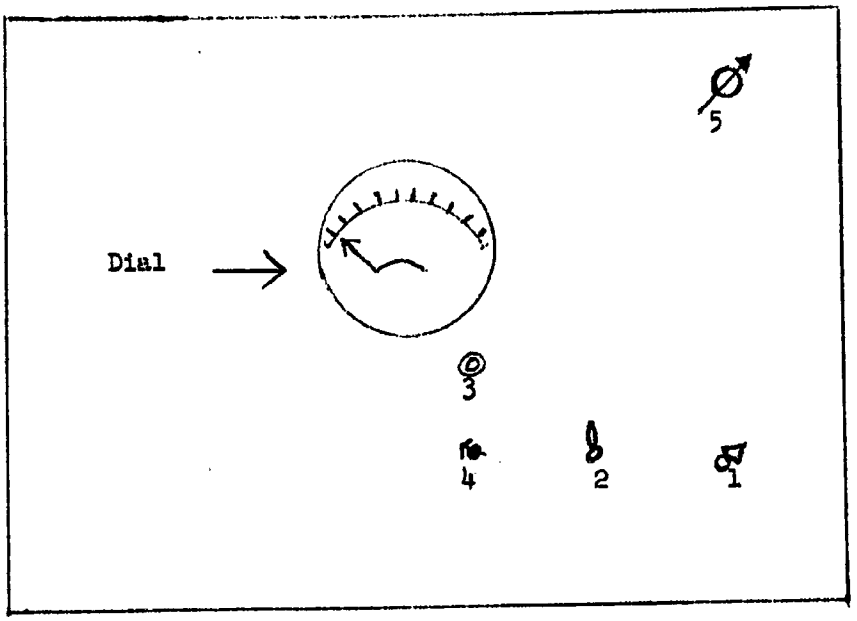

FIG. 1 Impact Sensitivity "Noisemeter" Control Box

8. Raise the hammer to 300 cm by turning the winch located at the left of the machine.

9. Place an inert sample in the center of a piece of one-inch square sandpaper and place it upon the cleaned anvil and gently set the striker on top of the sample.

10. Press switch No. 2 to return needle to zero and then release button at the right of the machine and observe where the needle comes to rest on the control panel. Adjust switch No. 5 to bring the needle to an average reading of 25 (5 small divisions) or as close as possible by dropping the hammer on five or more samples.

PREPARATION OF SAMPLE

1. Prepare about 5 grams of unground explosive that has passed a 20 mesh U. S. Standard screen and is retained upon an 80 mesh screen.

2. Select a cup that will measure a volume of powder equal to 35 mg ± 2 mg, or weigh out each sample if there is difficulty in maintaining the required tolerances.

3. Pelletize each sample into 3/16-in. diameter pellets using an Arbor press which is fitted with a torque wrench to indicate pressure applied. Pelletize about 25 samples. Check the weight of these pellets by weighing the total number of pellets and recording number and weight. (Any pelletizing machine, press or other device may be used as long as the pressure (30,000 psi) can be measured.) Be sure sample powder is level in the die prior to pressing. Only whole, unchipped samples are to be used in the 50% point determinations.

4. Place samples on soft paper in vacuum desiccator, run vacuum pump for 1/2 hour, close desiccator, turn off pump and leave for about 16 hours.

THE TEST

1. Cut several sheets of number 5/0 grade sandpaper into one-inch squares. Use sandpaper of 5/0 grit, A backing, 73 coating made by Abrasives Products, Inc., South Braintree, Mass.

2. In testing explosives, place sample as instructed under the section on turning on the machine and setting the listening device, item No. 9. Place shield in front of anvil. Run the hammer down to about 40 cm and push the release button on the right side of the machine. An explosion is indicated on the dial by a reading of 30 or more (6 small divisions). Determine an explosion point and a non-explosion point by dropping the hammer at various heights until an explosion is obtained using a fresh sample for each drop, then lowering the hammer the specified increment or increments until an explosion is not obtained. The dial, instead of being calibrated in cm, is calibrated in distances of 0.1 log units. Use increments of 0.1 log units and use increments of 0.1 log units regardless of hammer height. When the approximate height is found, record an explosion as a plus (+) and a non-explosion as a minus (-) on the data sheet. Run 20 samples, raising the hammer one increment if there is no explosion and continuing one increment at a time until an explosion occurs and then lowering the hammer similarly until a fresh specimen does not explode, then raising and lowering as required.

CALCULATION OF RESULTS

Results of the 20 test samples are recorded on the impact sensitivity test sheet, Fig. 2. The formula for obtaining the 50% point is given at the bottom of Fig. 2 with sufficient explanation for solving; however, a typical example will be explained. The calculations are made using either explosions or non-explosions whichever occur the least number of times. In the example there were 9 explosions and 11 non-explosions, therefore, the explosions were used. The table is filled in. The normalized height of the lowest line on which there is a test recorded is 1.7 (c). There was one explosion at the 1.7 level, 3 explosions at the 1.8 level, and 5 at the 1.9 level. The number N is the sum of the explosions (9 in this case).

The sum of the in_i is 13 (A); and the sum of the i^2n_i is 23 (B). Therefore m = median height is equal to $c + d\left(\frac{A}{N} \pm \frac{1}{2}\right)$; m = 1.7 + 0.1 $(1.44 - \frac{1}{2})$ = log 1.794 = 62 cm. The standard deviation is $1.62\ d\left(\frac{NB - A^2}{N^2}\right) + 0.029 = 0.08$.

PROCEDURE FOR TESTING EXPLOSIVE POWDER

If a test is made on molding powder of standard 20-80 mesh (U. S. Standard sieve sizes) material the preparation of the machine and of the sample is the same as in above except that the pelletizing step described above is omitted. The correct scoop is used (35 mg) or the material is weighed and the sample is carefully transferred volumetrically to the one-inch piece of sandpaper. The test and calculations are carried out as listed above.

ADDITIONAL INFORMATION

1. The anvil and striker are resurfaced after 10 shots with metallized explosives and 20 shots for non-metallized explosives.

2. Explosives are classed by impact sensitivity at the Naval Ordnance Laboratory as follows:

Ht, 50% cm	Class	Common explosives in this class
0-5	7	
5-10	6	
10-20	5	PETN
20-40	4	Tetryl, RDX
40-80	3	Comp B, torpex
80-160	2	HBX, TNT
160 and above	1	
No detonations	0	

3. When this procedure is used in conformance to explosives criteria, the sample used for measurements should be analyzed for composition.

ADDENDUM

The standard sample of Composition B--which should be designated by Holston numbers--is to be run at least three times a week in the pelletized form. A control chart is kept of the values and a mean established when any standard value is different from the mean by more than 3 sigma it will be assumed that the process is out of control and no samples will be run until the reason for the change has been found and/or corrected at least two successive standards will then be run to show that the process is under control.

IMPACT SENSITIVITY TEST Date_____

Identification of Sample_____ Pellet Wt._____ Mesh Size_____

Temperature_____ Humidity_____ Bldg. No._____

\+ = Explosion - = Non-Explosion

1.7^-	1.8^+	1.7^-	1.8^+	1.7^-	1.6	1.7^-	1.8^-	1.9^+	1.8^-
1.9^+	1.8^+	1.7^-	1.8^-	1.9^+	1.8^-	1.9^+	1.8^-	1.9^+	1.8^-

+

log	i	n_i	in_i	i^2n_i
1.7	0	1	0	0
1.8	1	3	3	3
1.9	2	5	10	20
	3			
	4			
		N = 9	A = 13	B = 23

i_o, i_1, i_2, etc., are arbitrary consecutive numbers set to denote the different log levels.

n_i denotes the number of explosions (or non-explosives) on the i_o, i_1, i_2, etc., lines.

N = sum of the +'s or -'s.
A = sum of the in_i.
B = sum of the i^2n_i.
c = normalized height of the lowest line on which there is a test recorded.
d = 0.1 log increment.

In formula (1) below, use + for explosion or - for non-explosion which ever occurs the least number of times.

If explosions use - 1/2. If non-explosion use + 1/2.

(1) $m = c + d \frac{A}{N} \pm 1/2$

$m = 1.7 + 0.1 (1.44 - 1/2) = 1.794.$

(2) $s.d. = 1.62\, d \sqrt{\frac{NB - A^2}{N^2}} + 0.029$

50% pt in cm = 62

Standard dev. = 0.08

References:

Formula (1) (Reference 2.)
Formula (2) (Reference 3.)

FIG. 2. Impact Sensitivity Test Sheet

VACUUM THERMAL STABILITY TEST

INTRODUCTION

The vacuum thermal stability test is a standard test for determining stability of explosives to storage conditions. This test is generally run at 100 to 120°C. The explosives are classed according to stability depending upon the quantity of gas evolved. (Reference 4.)

Thermal Stability Classes:

Vacuum thermal stability at 100°C	Vol gas/g/48 hr.
cc gas	Class
0 - 2	I
2 - 6	II
6 - 18	III
18 +	IV

Class I explosives are considered generally suitable for military use.

DESCRIPTION OF THE HEATING BLOCK

A circular aluminum block is drilled to hold 12 sample tubes (1/2 x 6-inch). A block is fitted with two sets of heating elements wound around the periphery of the block. One set has heavy windings and is used to heat the block to the desired temperature. It is controlled by a Fenwall thermo-regulator and one indicating pilot light. The second series of windings is much lighter and is used for intermittent control. Any control system may be used for the secondary winding as long as ± 0.2°C is maintained.

PREPARATION OF SAMPLE AND ASSEMBLY OF TUBES

1. VTS Test. The powder as it comes to the laboratory has been oven dried for 20 hours at 80°C. The sample prior to testing is placed in a vacuum desiccator and vacuum pulled for 1/2 hour at 0.5 - 1 ml of Hg; turn off pump, close desiccator and leave for about 16 hours.

2. The test tubes and capillary tubes are numbered 1 to 8. Each number on a test tube is used with its corresponding number on the capillary tube. Clean and dry as many test tubes as needed. (Run duplicates on all samples.) Weigh into each tube 5 grams ± 1.0 mg of dried sample. Wipe the ball and socket joint with a clean cloth. Rub a film of vacuum stop-cock grease on both joints. Using the capillary tube support, join the two together with a twisting motion to insure a good fit. Place a clamp over the joints and tighten sufficiently to make a good seal.

3. Evacuate the assembly by tipping the tube forward so that all the mercury runs into the cup leaving a path for the air to be exhausted from the sample. When a pressure of 0.25 to 0.5 mm of mercury is indicated by the vacuum manometer, the tube is placed in an upright position and the vacuum line is carefully disconnected.

4. Place the assemblies in the heated aluminum block. Leave for one hour.

5. After one hour, re-evacuate the tubes; place in block and read the capillary tube in cm from the top mark on the tube to the mercury level. Record. Record at the same time the barometric pressure in cm, and the room temperature in degrees Kelvin (273.2 + °C). Take reading twice a day until 48 hours has elapsed.

6. At the end of 48 hours, turn the machine off and clean the tubes. Calculate the gas evolved in ml/g/48 hr using the formula given below:

$$V_g = \frac{273.2\,(P_a + d - k)}{76\,w} \quad \frac{V_t - V_s}{t_o} + \frac{V_c + d\,L}{t_r}$$

where V_g = volume of gas in ml/g
P_a = atmospheric pressure (cm Hg)
d = length in cm of capillary tube (measured from a fixed point (see Fig. 3) at the top of capillary tube used in standardizing the tube volume) to the Hg level in the capillary tube
K = ht of Hg in cm from E to greatest height under vacuum
w = initial weight of sample in grams
V_t = volume of test tube (ml)
V_s = volume occupied by sample (calculated from its weight and density) in ml
t_o = temperature of the sample tube (bath temperature) in degrees Kelvin
t_r = temperature of capillary tube (room temperature) in degrees Kelvin
V_c = volume of capillary tube in ml from the sample tube to D (see illustration, Fig. 3)
L = volume per unit length of capillary tube (ml/cm)

7. This test is to be run at 120°C as well as 100°C for evaluating materials.

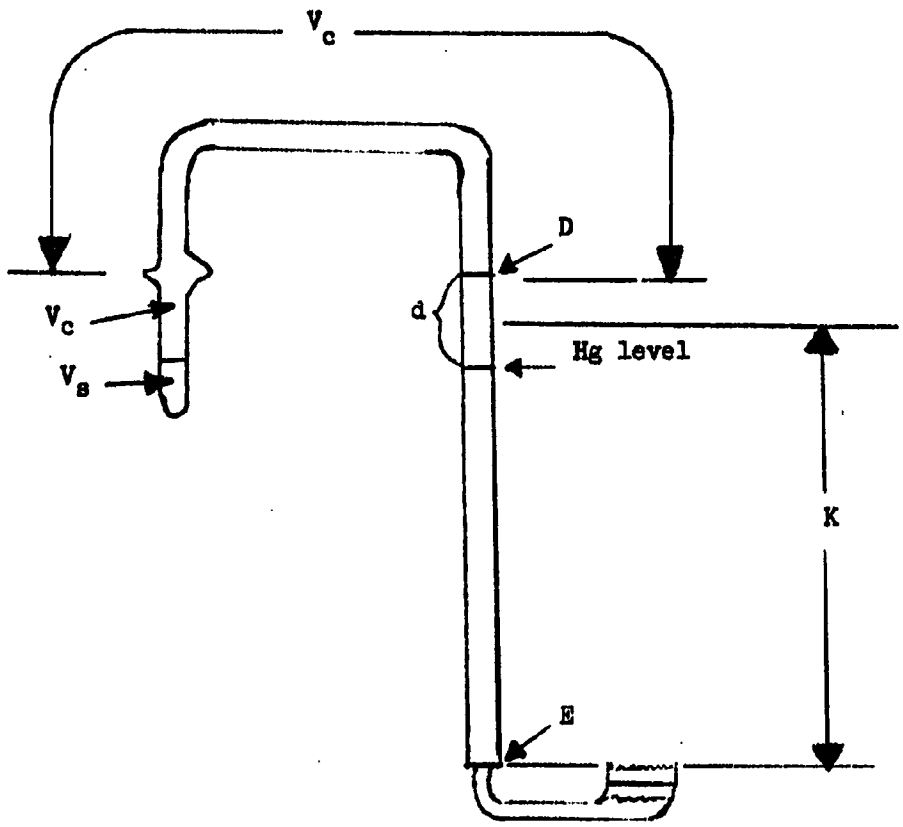

FIG. 3. Vacuum Thermal Stability (Sample Tube)

COMPRESSIVE STRENGTH TEST

THEORY

The compressive strength is calculated from a stress strain curve obtained while applying a load at a constant rate on a standard sample and from the dimensions of the sample. The modulus of elasticity is also obtained from this curve.

SAMPLE SPECIFICATIONS

The sides of the sample must be parallel and at right angles to the ends. The ends must be parallel. Use 1/2-in. x 3/4-in. specimens or 2-in. x 3-in. samples. Densities are always taken before test. Conduct test at temperature asked for on request slip. Most samples are at room temperature (70 to 80°C).

EQUIPMENT AND SUPPLIES NEEDED

1. A compression tester of suitable load range.
2. A recorder which will give the load vs. deflection curve.
3. A sample holder which will contain the sample both before and after rupture.

PROCEDURE

See standard procedure for the Baldwin Tester.

1. The sample is placed in the holder and then between compression plates.
2. The load is applied at a specified crosshead speed. The standard test uses a crosshead speed of 0.05 inches per minute.
3. After rupture the sample is removed from the holder and the pieces placed in a scrap box.

CALCULATIONS

1. The maximum compressive load in psi is calculated by dividing the maximum pounds at rupture by the area of the sample. The modulus of elasticity is calculated from the longest tangent line which can be drawn on the curve. See Fig. 4.

2. The total deflection to rupture is measured from the point where 100 psi is applied to the point where the stress-strain curve drops to a negative slope at rupture. The area under the curve is measured with a planimeter for three typical curves from each series of ten stress-strain curves to represent the energy to rupture. The area used is that between the axis and the curve extending over the length of the deflection to rupture.

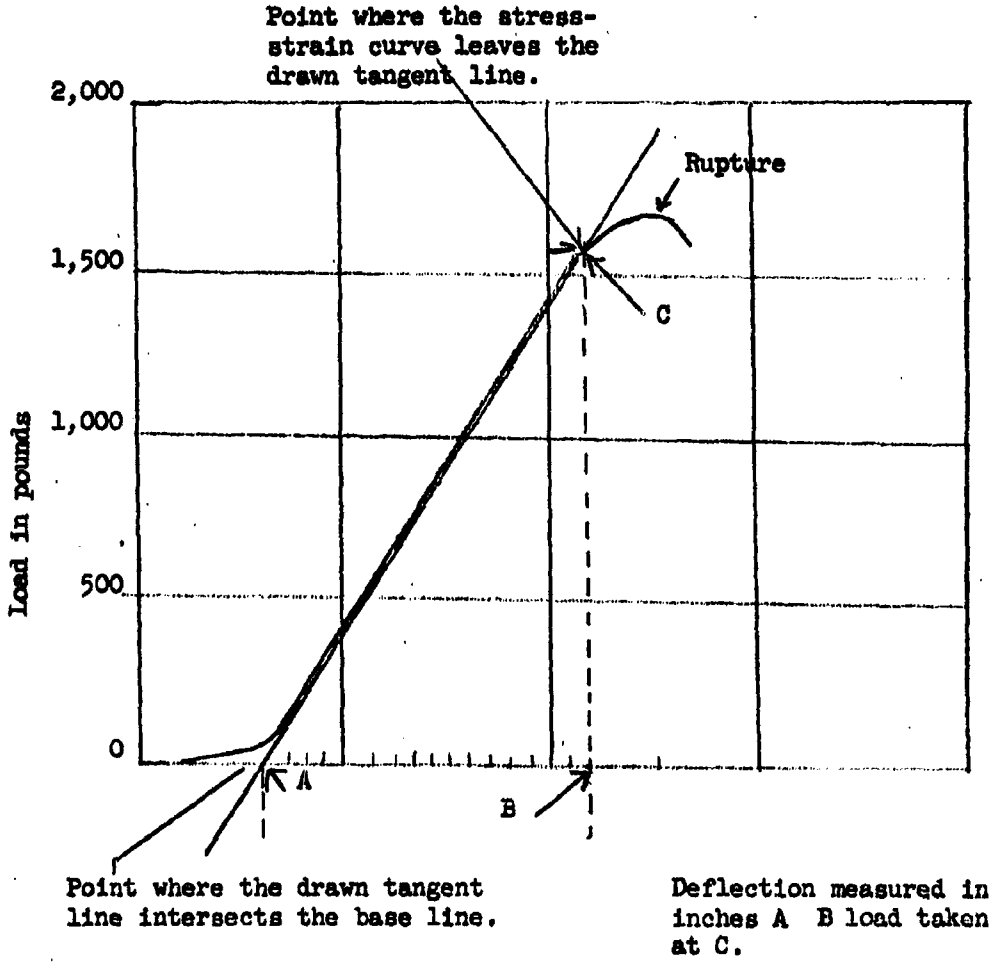

FIG. 4. Calculations for Modulus of Elasticity.

3. The modulus of elasticity may be calculated from this formula:

$$ME = \frac{A \times B}{C}$$

where ME = modulus of elasticity
A = the calculated psi at the point where the stress-strain curve leaves the drawn tangent line.[2]
B = height of sample in inches
C = deflection in (0.001) inches

MELTING POINT OF EXPLOSIVES

EQUIPMENT NEEDED

1. Melting point apparatus, Fisher Johns Cat. No. 12-142 (Fisher Scientific Company).
2. Round cover glasses 18 mm in diameter.
3. Nickel spatula.

PROCEDURE

1. Turn apparatus on and regulate the temperature so that the temperature rise is about 1°C every minute.

2. Place a small amount of sample between two cover glasses using the tip of the nickel spatula.

3. Note the temperature at which the material just begins to melt. Turn off the temperature control and note the temperature at which the sample just begins to crystallize. Report the average of the two. There will probably be about 2-3°C difference in the two readings.

4. For precise melting points on cast explosives such as TNT, use platinum resistance thermometer. This should not be used without prior instruction.

[2] If all replicate samples of the same material are of the same area, it is permissible to use a constant for A. Such a line can be drawn horizontally at the point where all samples fall within a range just above "A".

PERCENT MOISTURE DETERMINATION USING STERLING-BIDWELL MOISTURE TUBE

THEORY

This method works best on materials containing 1 to 5% moisture; however, it has been used successfully on PBX molding powders containing nylon with moisture content as low as 0.3% by increasing the sample size. It is used as a quick check to see if the materials comply with specifications. (Reference 5)

PROCEDURE

Connect together a 500 ml round-bottom flask (Note 1), a Sterling-Bidwell moisture tube similar to Braun No. 26344, and a condenser. Attach a drying tube to the top of the condenser to eliminate moisture pick-up from the atmosphere. The flask is heated by a heating mantle regulated by a variac.

Add 250 ml of the previously determined solvent (Note 2) together with a few boiling chips to the flask. Heat and allow the liquid to reflux until no more moisture collects in the tube (about 1 hour). Cool, disconnect the assembly keeping the flask stoppered. Wash the condenser and moisture tube with acetone and then dry.

If the sample has less than 0.5% moisture, add 40 grams of powder to the flask; if more add 25 to 30 grams. Reassemble the apparatus, reflux for three hours or until the water level in the moisture tube remains constant. Turn off the water in the condenser until the reflux level rises about half-way into the condenser. Resume the water flow and distill for an additional 5 minutes (Note 3). Record the number of milliliters of water collected in the moisture tube and calculate the percent water as follows:

$$\text{Percent water} = \frac{\text{ml of } H_2O \times 100}{\text{wt of samples in grams}}$$

NOTE 1. The flask, Sterling-Bidwell moisture tube and condenser must be clean and connected with ground glass joints only.

NOTE 2. The solvent should be less dense than water and insoluble in water. The following solvents are recommended: Tolune, B. P. 110°C; Xylene, B. P. 144°C.

NOTE 3. The heavy refluxing with the water turned off in the condenser will flush condensed moisture from the bottom of the condenser into the trap.

PROCEDURE FOR SHEAR STRENGTH OF EXPLOSIVES
(Similar to ASTM D732-46)

SCOPE

This method covers the punch type of shear test and is intended for use in determining the shear strength of test specimens of organic plastics or explosives in the form of molded or pressed disks in thicknesses from 0.250 to 0.500-inches.

DEFINITION

Shear strength is the maximum load required to shear the specimen in such a manner that the moving portion has completely cleared the stationary position. It is expressed in pounds per square inch based on the area of the sheared edge or edges.

APPARATUS

1. Testing Machine. The Baldwin testing machine of the constant-rate-of-crosshead speed type is used. The testing machine shall also be equipped with a load-indicating mechanism capable of showing the total compressive load carried by the test specimen.

2. Shear Tool. The shear tool of the punch type is constructed in this manner: The holder for the test specimen is a cup-type 2.00-inches in diameter by 1-1/2-inches high with a 0.500-inch hole drilled in the bottom. A piston type punch holder slightly smaller than the cup sets on top of the specimen. This receiver holds the 0.500-inch by 3.0-inch long punch. The receiver is held rigidly in place by three wing nuts. (See Fig. 5.)

TEST SPECIMEN

The specimen shall be a 2-inch diameter (approximately 0.250-inches thick) disk pressed into this form. The upper and lower surfaces shall be parallel to each other and reasonably flat (± 0.002-inches thick). If these tolerances can not be met on pressing, the specimen must be machined or discarded.

PROCEDURE

1. The test specimens are tested at room temperature unless otherwise specified and kept in a desiccator and specimen removed one at a time for testing.

2. At least five specimens shall be tested.

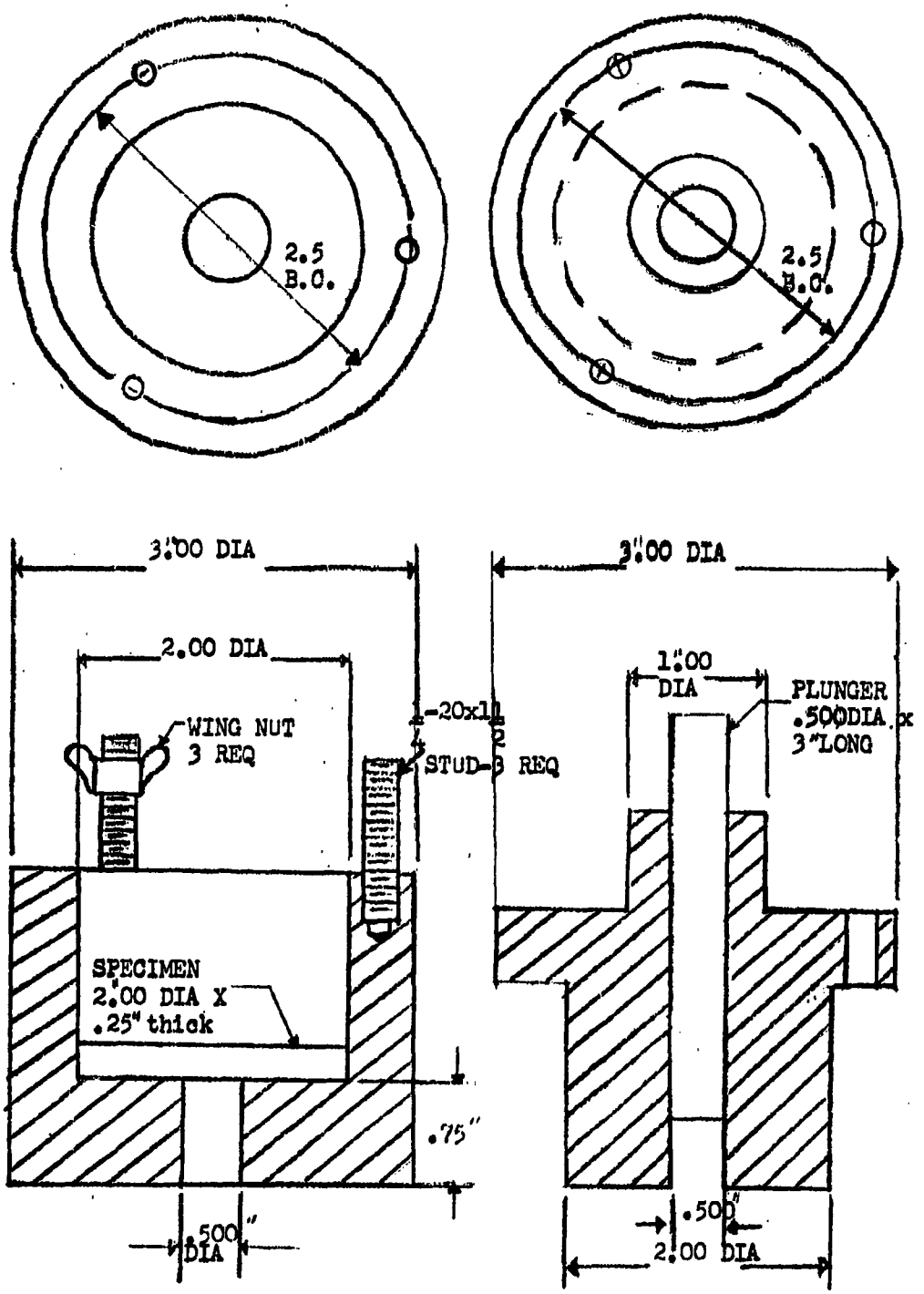

FIG. 5. Shearing Tool for 2-In. by 0.5-In. Thick Specimen.

SCALE 2.5" =3.0"

3. The specimen (after being accurately measured) is placed in the bottom of cup or specimen holder.

4. The punch holder is placed on top of the specimen and the three wing nuts tightened just enough to remove play but without cracking specimen.

5. Insert the 0.500-inch punch through the punch holder and until it rests upon the test specimen. Place the assembly under the crosshead beam.

6. The crosshead speed of the machine during the test is 0.05-inch per minute measured when the machine is running idle.

7. The punch is pushed down far enough to indicate on the load-indicating mechanism that the total load needed to shear the sample has been achieved.

REPORT THE FOLLOWING

1. Complete identification of the specimen.
2. Temperature of the test.
3. Humidity of the room at time of test.
4. Conditioning procedures used if different from normal testing.
5. The calculated shear strength in psi is determined by dividing the load by the area of the sheared edge which shall be taken as the product of the thickness of the specimen by circumference of the punch. Report individual and average values.

PROCEDURE FOR MODULUS OF RUPTURE BY SIMPLE BENDING

DESCRIPTION OF EQUIPMENT

The test utilizes the principle of simple bending on specimens 3-1/4-inch long by 0.3-inch wide and 0.2-inch thick. However, the original Aminco modulimeter had to be modified in order to be used on explosive compounds having very low elastic limits. The original modulimeter was used mainly to test the modulus-of-elasticity of metal specimens. Figure 6 is a drawing of the modified modulimeter.

PREPARATION OF TEST SPECIMENS

The test specimens are 3-1/4-inch long, 0.3-inch wide and from 0.2 to 0.3-inch in thickness. This thickness is important and may not vary more than 0.005-inch from end to end. A leveling devise may have to be made to insure the correct size samples.

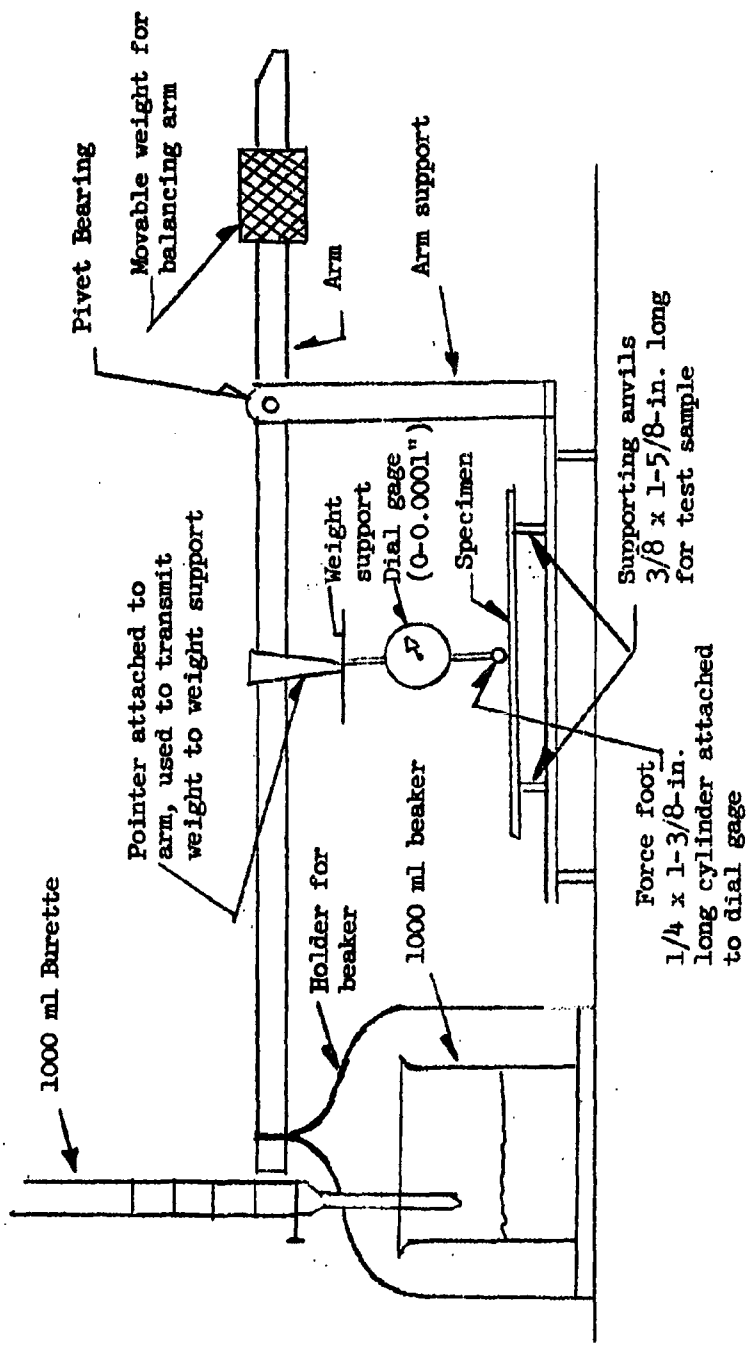

FIG. 6. Modified Aminco Modulimeter.

TEST PROCEDURE

1. Remove the beaker from the holder. Clean, dry, and replace in holder.

2. Fill the 1000 ml burette to the mark with water and check the flow at 100 ml per minute. Use a piece of rubber tubing fitted to the tip of the burette fitted with a punch clamp.

3. Set the supporting anvils at 3.00 inches, being sure that the force-foot is in the middle (1-1/2 inches from each end).

4. Place specimen on the anvil shoulders centering the specimen on the holders so that the center is directly under the center of the force-foot.

5. Balance the arm by adjusting the movable weight just enough to indicate a slight movement of the dial gage.

6. Adjust the dial gage to zero.

7. Start the flow of water into the beaker by releasing the pinch clamp.

8. Stop the flow of water at exactly the same time the sample ruptures by using either the pinch clamp or the thumb and forefinger.

CALCULATIONS

1. The modulus of rupture is calculated as follows:

$$MR = \frac{27 \times wt}{2 \times w \times h^2}$$

where MR = modulus of rupture
wt = weight of water in lb (0.0022 x ml of water)
w = width of sample in inches
h = height of sample in inches

REFERENCES

1. OSRD 5744, Physical Testing of Explosives, Part 2.

2. AMP Report No. 101-1R, Statistical Analysis for a New Procedure in Sensitivity Experiments.

3. Dixon and Massey, Introduction to Statistical Analysis.

4. OSRD 787, Progress Report on Chemical Tests for New Explosives.

5. Moisture Content of Powders, Handbook of Plastics, by Simonds and Ellis. p 61.

ABSTRACT CARD

U. S. Naval Ordnance Test Station
Standard Laboratory Procedures, Part 1. Stability, Sensitivity, Physical Properties and Analyses for High Explosives, by O. K. Pennington. China Lake, Calif., NOTS, August 1961. 26 pp. (NAVWEPS Report 7764, Part 1, NOTS TP 2742), UNCLASSIFIED.

ABSTRACT. The Standard Laboratory Procedures for explosives herein reported are those developed for use by the Explosives and Pyrotechnics Division of the Propulsion Development Department. This part

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